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Halogen-free flame retarded cold-mix epoxy asphalt binders: Rheological, thermal and mechanical characterization



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HIGHLIGHTS

• Halogen-free flame retarded cold-mix epoxy asphalt binder (CEAB) has been developed.

• The flame retardancy of the neat CEAB is significantly increased by halogen-free flame retardant.

• The addition of halogen-free flame retardants improves the glass transition temperature, thermal stability and tensile strength of the neat CEAB.

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ABSTRACT

Fire safety of asphalt mixtures in highway tunnels has become a problem of great concerns since asphalt and polymer modified asphalt binders are quite flammable and tend to release poisonous gases and smoke while burning. Therefore, flame retardants are incorporated into asphalt binder to improve the fire retardancy of asphalt mixtures. In this paper, aluminum trihydroxide (ATH) and zinc borate (ZB) were used as halogen-free flame retardants to prepare flame retarded cold-mix epoxy asphalt binders (CEABs). The addition of halogen-free flame retardants increased flame retardancy of the neat CEAB. In addition, ATH-ZB retarded CEAB had higher limited oxygen index (LOI) than single ATH and ZB retarded CEABs at the same content. The presence of halogen-free flame retardants increased the viscosity of the neat CEAB. The viscosity of ATH/ZB retarded CEAB was lower than that of ATH retarded CEAB at a specific content. The incorporation of halogen-free flame retardants improved by the halogen-free flame retardants. ATH/ZB retarded CEAB had better thermal stability than ATH retarded CEAB at the same content. The addition of halogen-free flame retardants increased the tensile strength of the neat CEAB. Uniform dispersion of halogen-free flame retardants increased the tensile strength of the neat CEAB. Uniform dispersion of halogen-free flame retardants in CEABs was observed.

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1. Introduction

Rigid pavements of Portland cement concrete (PCC) and flexible pavements of asphalt mixtures are currently known and used road pavements for tunnels [1]. It is well accepted that PCC has a higher fire resistance and thermal stability and very low emission of toxic gases, which assures the evacuation of people and the transit of vehicles in the event of fire. However, PCC pavements contain difficulties and defects in paving and maintenance, resulting in inadequate performances with regards to functional characteristics. In addition, compared to the asphalt mixtures, the costs of PCC pavements for construction and subsequent maintenance are much higher. Thus, asphalt mixtures have been widespreadly used in

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https://doi.org/10.1016/j.conbuildmat.2018.08.018 0950-0618/© 2018 Elsevier Ltd. All rights reserved. tunnels due to their excellent surface evenness, skid resistance and ease of application. However, as a mixture of hydrocarbons. asphalt is flammable. When used in tunnel construction, asphalt can be ignited at less than 300 °C and release toxic smoke and heat. Thus, the development of flame retarded asphalt binders with adding organic and inorganic flame retardants (FRs) has become a preferable solution to improve the fire retardancy of flexible pavements in tunnels [2–4]. Among all organic FRs, brominated flame retardants (BFRs) have been widely used in asphalt mixtures [5,6]. Due to concerns about the persistence, bioaccumulation, and toxicity (PBT), some BFRs, such as brominated diphenylethers (BDEs) and polybrominated biphenyls (PBBs) halogenated flame retardants have been banned to use in the last decade [7]. Hence, the use and demand of halogen-free flame retardants to substitute BFRs have grown rapidly [8]. As the main halogen-free flame retardants, mineral fillers, especially aluminum trihydrate (ATH) and magnesium hydroxide (MH), have attracted increasingly interest for asphalt binders [9–12].

Epoxy asphalt (EA) is a thermosetting polymer modified asphalt, which is mainly made up of epoxy monomer, curing agent and asphalt [13–15]. Epoxy asphalt binder can be classified into three types according to mixing temperature: hot-mix epoxy asphalt binder (HEAB, 160-190 °C), warm-mix epoxy asphalt binder (WEAB, 110–140 °C) and cold-mix epoxy asphalt binder (CEAB, ambient temperature) [16–18]. When epoxy monomer and curing agent are mixed together, an irreversible cure reaction will take place and form three dimensional crosslinking networks, in which asphalt molecules are restricted [19–22]. As a result, thermoplastic asphalt turns to thermosetting elastomer, endowing epoxy asphalt mixtures with high strength and toughness, good fatigue resistance, skid resistance and temperature stability and long durability [23,24]. Epoxy asphalt binder has been widely used not only on the steel bridge decks, especially in China, but also in tunnels, on airfields and porous asphalt pavements with high durability and at intersections of heavy duty roads [25,26]. However, epoxy asphalt is also quite combustible and produces much smoke and toxic gases since both asphalt and epoxy resin is intrinsically flammable [27]. It will be hazard to pave non-flame-retarded epoxy asphalt mixtures in tunnels. Consequently, to improve the fire resistance of epoxy asphalt binder is of great importance.

In present paper, ATH and ATH combined with zinc borate (ZB) were used as halogen-free flame retardants to improve the flame retardancy of cold-mix epoxy asphalt binder. Oxygen index instrument, Brookfield rotational viscometer, differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), tensile test and scanning electron microscopy were employed to determine the influence of halogen-free flame retardants on the flame retardancy, viscosity, glass transition temperature (Tg), thermal stability, mechanical properties and morphology of cold-mix epoxy asphalt binders.

2. Experimental

2.1. Materials

Cold-mix epoxy asphalt binder contains two components: epoxy monomer and asphalt with curing agent. Epoxy monomer is diglycidyl ether of bisphenol A (DGEBA). An amine cure agent (NDB196B) was prepared by our laboratory. ATH is white powder with Al(OH)₃ content more than 99.4%. Its average particle size is 1250 mesh. Zinc borate (2ZnO-3B₂O₃-3.5H₂O) was also white powder with average particle size of less than 7 μ m. The weight ratio of ATH/ZB is 3:1.

2.2. Samples preparation

DGEBA and NDB196B were heated at 60 °C and mixed a 250 mL beaker at 2000 rpm for 5 min. Then, ATH, ZB or ATH/ZB was introduced into the CEAB mixture and agitated using Fluko FM300 high-shear emulsifier (Shanghai, China) at 3000 rpm at 5 min. Finally, the mixture was cured for 2 days at 60 °C in Toflon molds. The weight percents of ATH, ZB or ATH/ZB in CEABs were 10, 20 and 30, respectively.

2.3. Tests and measurements

Limited oxygen index (LOI) of flame retarded CEABs were tested with HC-2C oxygen index instrument (China) on the sheets of $100~mm\times10~mm\times4~mm$ according to ASTM D2863.

Rotational viscosities of the neat CEAB and flame retarded CEABs were measured in a Brookfield rotational viscometer with spindle 28 (NDJ-1C, China) under the guideline of ASTM D4402. The rotational viscosity began to be recorded when all components of samples were stirred at 3000 rpm at 5 min.

The T_gs of the neat CEAB and flame retarded CEABs were evaluated by differential scanning calorimetry (DSC) (Mettler Toledo DSC 1, Leicester, UK). The DSC measurements were carried out from -20 to 80 °C at a ramp rate of 20 °C/min under a nitrogen purge.

Thermal stabilities of the neat CEAB and flame retarded CEABs were determined by a Mettler Toledo TGA/DSC 1 Star^e system (Leicester, UK). The test was performed from 50 to 600 °C with a heating rate of 20 °C/min under a nitrogen purge.

Mechanical properties of the neat CEAB and flame retarded CEABs were performed by an Instron 3366 universal test machine (Norwood, MA, USA) according to ASTM D638. At least five samples were measured with a strain rate of 200 mm/min.

The morphology of the neat CEAB and flame retarded CEABs was observed by a field-emission scanning electron microscopy (FE-SEM). The sample was surface fractured in the liquid nitrogen and coated with gold before SEM observations.

3. Results and discussion

3.1. Flammability

LOIs of the neat CEAB and flame retarded CEABs are presented in Fig. 1. The LOI of the neat CEAB is 20.0%, indicating that CEAB is very flammable. Obviously, the addition of flame retardants increases the LOI of the neat CEAB. Additionally, the LOI of ATH and ZB retarded CEABs increases with the increase of flame retardant contents. At a specific content, the LOI of ZB retarded CEAB is slightly higher than that of ATH retarded CEAB. However, the LOIs of both ATH and ZB retarded CEABs are lower than that of ATH/ZB retarded one at the same content. This indicates ZB has synergistic effect on the flame retardancy of ATH in the neat CEAB. Similar results were also reported in ATH/ZB modified rigid poly (vinyl chloride) (PVC) [28]. For ATH/ZB retarded CEABs, LOIs of all ATH/ZB modified CEABs are greater than 23%. CEAB with

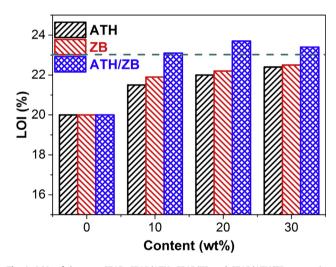


Fig. 1. LOIs of the neat CEAB, CEAB/ATH, CEAB/ZB and CEAB/ATH/ZB composites.

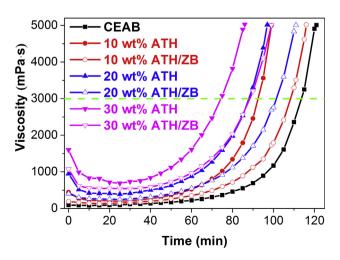


Fig. 2. Viscosity versus time curves of the neat CEAB and flame retarded CEABs at 60 $^\circ\text{C}$.

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